

Sampling statistics applied to automated TAPPI dirt counting

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ABSTRACT: *There is considerable variation in pulp dirt count from handsheet to handsheet within a stock sample. The effect of sample size on dirt count test precision is not well understood throughout the paper industry. The author presents basic statistical techniques which allow testers to form precision statements for dirt count test results and to determine the sample size needed to obtain a given precision. Examples illustrate the statistical concepts.*

KEYWORDS: *Dirt count, measurement, precision, sampling, statistical analysis, statistics, TAPPI, test methods.*

The recent proliferation of devices available for automated dirt counting of pulp and paper samples has greatly improved the efficiency of this task. As more mills introduce recycled fiber into their systems, dirt level becomes a critical pulp quality specification. Automated dirt counters are in common use in many mills and laboratories throughout the world. It has been our experience that the effect of sample size, i.e., number of handsheets tested per measurement group, on dirt count result precision does not have wide understanding.

The use of an automated instrument virtually eliminates the substantial operator error present when manually using TAPPI Test Method

T 213. No instrument can compensate for sample-to-sample variation within a population, however. TAPPI Test Method T 213 suggests sampling at least ten–1500 cm² sheets for each dirt count measurement on lap pulp. Measuring both sides of each sheet would encompass 3 m² of surface area per dirt count measurement. The method suggests a sample size of at least 4000 cm² surface area when measuring handsheets or pads made from slush pulp (1).

Our primary interest in dirt counting is to evaluate the effectiveness of deinking and contaminant control programs both in the laboratory and in mills. We have commonly seen dirt count results expressed to the nearest 1 or 0.1 ppm area only to

find that the stated result relied upon measurement of a single handsheet. Comparison of dirt count results without an estimate of the measurement precision may lead to erroneous conclusions. A laboratory study determined the relationship between sample size (number of handsheets) and the precision for stating dirt counts.

Experimental

Deinked pulp samples were prepared in our laboratory pilot plant. A sorted white ledger recovered paper furnish was repulped and chemically treated to aggregate and densify the laser ink particles. Earlier investigators have previously reported the details of this process (2). A number of different centrifugal forward cleaner cone configurations were evaluated for their ability to remove the aggregated ink. Composite 5-g pulp samples were obtained from each cleaner's accept pulp. Ten 3-g Noble and Wood (8 in. x 8 in.) handsheets were made from aliquots randomly drawn from each 5-g pail while continuously stirring. We have found that the 73 g/m² basis weight of these sheets is heavy enough to avoid showthrough of dirt particles from one side to the other when viewed with reflected light.

Dirt counting was performed with an ASA 2000 Sheet Analyzer. This is a scanner-based system which uses reflected light and a document scanner to measure the entire sheet sur-

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face area. The 400 dots/in. pixel size allows definition of dirt particles down to a minimum of 0.015 mm². This is well below the 0.04 mm² TAPPI dirt count minimum size cut-off (3). The system pixel size is calibrated using a dot target of circles of known areas. The measurement is calculated to exclude particles smaller than the TAPPI minimum size cutoff from the dirt count tabulation.

Each side of each 10-sheet sample was measured, and the individual side's TAPPI dirt count recorded. Any ink in the sample is seen as a black speck against a high brightness background providing high contrast for the measurement. Previous internal work had determined that the dirt count of the top side was independent of the wire side of a handsheet within a given pulp source. The two sides of the handsheet can then be considered as independent samples. Twenty independent random measurements were then recorded for each pulp source. The total sheet surface area measured for each pulp source was about 0.7 m².

Statistical analysis

The objective of the statistical inference is to predict the true average dirt count of the pulp (population mean) based upon the average dirt count of the sample (sample mean). Each sample mean and variance were calculated by the following standard formulae:

$$\bar{x} = (\sum x_i)/n \quad (1)$$

$$S^2 = [\sum x_i^2 - (\sum x_i)^2/n]/(n-1) \quad (2)$$

where

x = individual measurement value

n = sample size

S² = sample variation.

The statistics of simple random sampling were used to compute the standard error and 95% confidence limits about each mean (4). Formation of handsheets is sampling without re-

I. Instrument repeatability for 20 repeated measurements on a single handsheet side using pulp source B

TAPPI dirt, ppm			
8.82	9.03	8.82	8.82
9.14	8.60	8.49	8.39
8.71	8.92	8.49	8.39
9.03	8.82	8.49	8.28
8.71	9.03	8.28	8.39
n = 20			
mean = 8.68 ppm			
sample variance = 0.07			
sample standard deviation = 0.27 ppm			

II. Twenty measurements on different handsheet sides from pulp source B

TAPPI dirt, ppm			
13.79	3.71	4.40	7.30
6.84	18.31	5.68	15.76
12.86	5.33	10.43	7.07
8.28	7.53	17.15	11.82
30.59	5.45	12.15	4.98
n = 20			
mean = 10.47 ppm			
sample variance = 41.88			
sample standard deviation = 6.47 ppm			

placement. If we assume that the population (pulp supply) is very large compared to the sample being drawn, the standard error of the mean can be easily calculated by the following formula:

$$S_{\bar{x}} = (S^2/n)^{1/2} \quad (3)$$

The student's t distribution can then be used to calculate a confidence interval about the mean. The t tables are in the above text (4) and in most other standard statistical texts and references. For a 95% confidence interval, the tabular t-value for 19 degrees of freedom (sample size minus one) is 2.093. The t values for large samples at 95% confidence are approximately equal to 2. The confidence interval about the mean is then plus or minus the tabular t-value times the standard error. The mathematical expression for this is $\bar{x} \pm tS_{\bar{x}}$. A 95% confidence about the

mean states that there is only a 5% probability that the true population mean falls outside the stated interval around the sample mean.

It is usually desirable to know in advance what sample size is necessary to produce a given measurement precision. In a population with a given variation, increasing the sample size improves the precision of the estimate. If we specify a given measurement error (or precision) of $\pm E$ ppm, we obtain the following equation:

$$E = tS_{\bar{x}} = t(s^2/n)^{1/2} \quad (4)$$

Mathematically rearranging to solve for n, the sample size, provides the following equation:

$$n = (t^2 s^2 / E^2) \quad (5)$$

In this case, n is the number of handsheet side measurements to

III. Results of TAPPI dirt count, ppm area, statistical analysis

Pulp source	Mean	Range, low-high	Std. error	Lower confidence limit (95%)	Upper confidence limit (95%)
A	9.38	4-28	1.34	6.58	12.18
B	10.47	4-31	1.45	7.44	13.50
C	8.89	0-40	2.16	4.37	13.41
D	12.85	3-33	1.60	9.51	16.19
E	7.26	0-16	0.94	5.29	9.23

IV. Number of handsheet sides required to achieve a specified error

Error about mean @ 95% confidence:					
	± 1 ppm	± 2 ppm	± 3 ppm	± 4 ppm	± 5 ppm
Number of handsheet side measurements required:					
Pulp source					
A	156	39	17	10	6
B	183	46	20	11	7
C	408	102	45	26	16
D	223	56	25	14	9
E	78	20	9	5	3
Mean (all samples)	210	53	23	13	8

achieve a measurement of the given precision. Note that the required sample size varies inversely with the square of the desired precision. Keep in mind that to apply the above formulae, a good estimate of the variance must be known in advance. It is essential that sampling must be performed randomly and without bias for these equations to be applied properly. The appendix to this paper includes a detailed example explaining how to perform these statistical calculations.

Instrument repeatability vs. error due to sampling

Testing error can be due to population variability. It can also be due to the repeatability of the test equipment itself. It is useful to partition the error in terms of variance between these two sources. A single

handsheet side was repeatedly measured 20 times. **Table I** contains the results. The sample had a mean of 8.68 ppm. The high measurement was 9.03 ppm, and the low measurement was 8.28 ppm. The variance due to instrument error was 0.07.

Twenty handsheet sides from the same pulp source were then measured. **Table II** contains the results. The sample mean was 10.47 ppm. Individual test results ranged from 3.71 ppm to 30.59 ppm. The variance due to combined instrument and sampling error was 41.88. From this data, we estimate that only 0.2% (0.07/41.88) of the observed variance is due to the instrument itself, and 99.8% of the variance is due to population variability. For practical purposes, all the observed variation could be attributed to sampling.

Results

Table III contains the results of the statistical analysis. The means of the five samples ranged from a low of 7.3 to a high of 12.8 ppm TAPPI dirt. Individual dirt concentration measurements within a single pulp source were quite variable. In the most extreme case, individual measurements ranged from 0 to 40 ppm. The error of the estimate ranged from ± 2.0 ppm for sample E to ± 4.5 ppm for sample C.

Table IV reports the number of handsheet sides required for a given measurement precision. Depending upon the sample variability, a measurement precision of ± 1 ppm would require testing of 78–403 handsheet sides. This intensity of testing is certainly beyond the capabilities of most laboratories. A measurement precision of ± 3 ppm required 9–45 handsheet sides with an average requirement of 23 sides.

We have adopted a standard measurement size of 20 handsheet sides with a stated measurement precision of ± 3 ppm for future evaluations at this approximate dirt level. If the measurement precision for a particular application is very critical, we repeat this procedure for each pulp sample of interest.

Effect of dirt level

Our experience has shown that as the dirt content of the pulp increases the absolute precision (in terms of ppm area) of the measurement will decrease at constant sample size. Higher dirt levels will generally lead to poorer absolute precision. The relative precision (in terms of percentage of the mean dirt count) will generally improve, however. For example, 20 handsheet sides at a mean dirt count of 10 ppm would yield a measurement precision of ± 3 ppm or 30% of the mean. Measurement of 20 handsheet sides at a mean dirt count of 100 ppm gave a measurement precision of ± 18 ppm—6 times

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greater than the cleaner sample. The relative precision improved to $\pm 18\%$ of the mean, however. The reverse relationship is generally true for very clean pulp samples.

Summary

Any evaluation of trial results or normal mill operating data should contain an estimate of the measurement precision. Dirt count values are critical quality specifications for recycled fiber systems. If test results are stated without an understanding of the measurement precision, false and potentially misleading conclusions may result. Due to the natural variation in pulp dirt level from any given source, a relatively large sample size will probably be necessary to achieve a reasonable measurement precision. The techniques described above should allow the measurement precision to be determined easily for a variety of testing applications. ■

Literature cited

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The author acknowledges the contributions of Jan Kinnaly and Cynthia Olson to the laboratory evaluation. Thanks are also due to Jeff Marcantonio of Applied Vision Systems for his review of the manuscript.

Received for review July 27, 1993.

Accepted Dec. 28, 1993.

Appendix

Sample calculation using pulp source B data from Table II

$$\sum x_i = (13.79 + 3.71 + \dots 4.98) = 209.43$$

$$(\sum x_i)^2 = (209.43)^2 = 43860.92$$

$$\sum x_i^2 = (13.79^2 + 3.71^2 + \dots 4.98^2) = 2988.78$$

$$n = 20$$

$$\text{Sample mean} = \bar{x} = \sum x_i / n = 209.43 / 20 = 10.47$$

$$\text{Sample variance} = s^2 =$$

$$[\sum x_i^2 - (\sum x_i)^2 / n] / (n - 1) =$$

$$[2988.78 - (43860.92 / 20)] / 19 = 41.88$$

Note that many inexpensive scientific calculators will produce the above basic statistical parameters.

$$\text{Standard error of mean} = S_{\bar{x}} = (s^2 / n)^{1/2} =$$

$$(41.88 / 20)^{1/2} = 1.45$$

Tabular t value, 95% confidence, α of 0.025¹, degrees of freedom = $n - 1 = 20 - 1 = 19$

$$t = 2.093$$

Measurement precision with 95% confidence = $\pm t S_{\bar{x}} = \pm (2.093)(1.45) = \pm 3.0$ ppm

$$\text{Lower 95\% confidence limit} = \bar{x} - t S_{\bar{x}} = 10.5 - 3.0 = 7.5 \text{ ppm}$$

$$\text{Upper 95\% confidence limit} = \bar{x} + t S_{\bar{x}} = 10.5 + 3.0 = 13.5 \text{ ppm}$$

To achieve a specified error of ± 5 ppm:

$$n = t^2 s^2 / E^2 = [(2.093)^2 (41.88)] / 5^2 = 7.34$$

Approximately 7 handsheet sides are necessary.

To achieve a specified error of ± 1 ppm:

$$n = t^2 s^2 / E^2 = [(2.093)^2 (41.88)] / 1^2 = 183.46$$

Approximately 183 handsheet sides are necessary.

¹Most t-tables show the t-value as a function of α , the area in each tail of the two-tailed probability distribution which is outside the confidence interval, i.e., $1 - (0.025 + 0.025) = 0.95$ probability of the true population mean falling within the interval.